

[TDC Form  
Similar to: Form PTO-1390]

U.S. DEPARTMENT OF COMMERCE  
PATENT AND TRADEMARK OFFICE

JCO8 Rec'd PCT/PTO 08 FEB 2001

ATTORNEY'S DOCKET NUMBER  
43050

**TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371**

U.S. APPLICATION NO. (if known, see 37 C.F.R. 1.5)  
**09/762545**

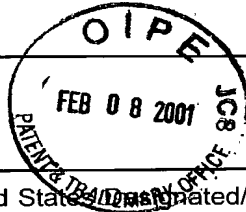
INTERNATIONAL APPLICATION NO.  
PCT/US99/19458 ✓

INTERNATIONAL FILING DATE  
26 August 1999 ✓

PRIORITY DATE CLAIMED  
26 August 1998 ✓

TITLE OF INVENTION  
**INTERNAL MOLD RELEASE FOR LOW DENSITY REACTION INJECTION MOLDED POLYURETHANE FOAM**

APPLICANT(S) FOR DO/EO/US  
Ron H. Niswander ✓



Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
  - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☐ has been transmitted by the International Bureau.
  - c. ☒ is not required, as the application was filed in the United States receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
  - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☐ have been transmitted by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the Annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

**Items 11. to 15. below concern other document(s) or information included:**

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ A FIRST preliminary amendment.  
☐ A SECOND or SUBSEQUENT preliminary amendment.
13. ☐ A substitute specification.
14. ☐ A change of power of attorney and/or address letter.
15. ☐ Other items or information:

U.S. APPLICATION NO. (If known, see 37 C.F.R. 1.50) <div style="font-size: 1.5em; font-weight: bold; text-align: center;">09/762545</div>	INTERNATIONAL APPLICATION NO. <div style="font-weight: bold;">PCT/US99/19458</div>	ATTORNEY'S DOCKET NUMBER <div style="font-weight: bold;">43050</div>
--	---	---

17. <input checked="" type="checkbox"/> The following fees are submitted: <div style="margin-left: 20px;"> <b>Basic National Fee (37 CFR 1.492(a)(1)-(5)):</b>            Search Report has been prepared by the EPO or JPO . . . . \$ <b>860.00</b>            International preliminary examination fee paid to            USPTO (37 CFR 1.482) . . . . . \$ <b>690.00</b>            No international preliminary examination fee paid to            USPTO (37 CFR 1.482) but international search fee            paid to USPTO (37 CFR 1.445(a)(2)) . . . . . \$ <b>710.00</b>            Neither international preliminary examination            fee (37 CFR 1.482) nor international search fee            (37 CFR 1.445 (a)(2)) paid to USPTO . . . . . \$ <b>1,000.00</b>            International preliminary examination fee paid to            USPTO (37 CFR 1.482) and all claims satisfied provisions            of PCT Article 33(2)-(4) . . . . . \$ <b>100.00</b> </div> <div style="text-align: right; margin-top: 10px;"> <b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b> </div>	CALCULATIONS	PTO USE ONLY
Surcharge of \$ <b>130.00</b> for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)). <span style="float: right;">+</span>	\$ <b>860.00</b>  \$ <b>130.00</b>	

Claims	Number Filed	Number Extra	Rate		
Total Claim	9 - 20 =	0	X \$ <b>18.00</b>	\$ <b>0.00</b>	
Independent Claims	2 - 3 =	0	X \$ <b>80.00</b>	\$ <b>0.00</b>	
Multiple dependent claim(s) (if applicable)			\$ <b>0.00</b>	\$ <b>0.00</b>	

Processing fee of \$ <b>0</b> for furnishing the English Translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)). <span style="float: right;">+</span>	\$ <b>0.00</b>	
<b>TOTAL NATIONAL FEE =</b>		
		Amount to be refunded: \$
		charged: \$

a. ☐ A check in the amount of \$\_\_\_\_\_ to cover the above fees is enclosed.

b. ☒ Please charge my Deposit Account No. **04-1512** in the amount of \$ **990.00** to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. **04-1512**. A duplicate copy of this sheet is enclosed.

Note: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO: <b>Graham E. Taylor</b> <b>The Dow Chemical Company</b> <b>Intellectual Property</b> <b>P.O. Box 1967</b> <b>Midland, Michigan 48641-1967</b> <b>UNITED STATES OF AMERICA</b>  Phone: <b>517-636-7393</b>	Signature: <u><i>Shelley D. Storey</i></u> <div style="text-align: center;"> <b>Shelley D. Storey</b>, Registration No. <b>45,135</b> </div> Date: <u>08 February 2001</u>
---	---

INTERNAL MOLD RELEASE FOR LOW DENSITY REACTION  
INJECTION MOLDED POLYURETHANE FOAM

FIELD OF THE INVENTION

This invention relates to molded foams. More particularly, it relates to a method for preparing a molded polyurethane foam article using an internal mold release composition.

5

DESCRIPTION OF THE PRIOR ART

Molded polyurethane foams (hereinafter "molded foams") are useful in a variety of applications. For example, molded foams are useful in the construction of furniture, automobiles, and buildings. Molded foams are especially important because they are lightweight and resistant to moisture, weather, temperature extremes, and aging.

10

Molded foams are produced from foamable reaction mixtures. A foamable reaction mixture minimally contains of an organic polyisocyanate, an active hydrogen containing compound, a catalyst, and a blowing agent.

15

To obtain a molded foam article, a foamable reaction mixture is injected into a mold, wherein the mixture foams and then solidifies in a compacted state. The mixture takes on the mold's shape and features. Injection molding or reaction injection molding (RIM) processes can be used to prepare the molded foam article.

To facilitate removal of the molded foam article from the mold, an external mold

release (EMR) agent can be used to coat the mold. Examples of typically used EMR agents are waxes, soaps, and oils. The EMR agent reduces the mold opening force required to free the molded foam article from the mold.

Unfortunately, the use of EMR agents can have deleterious effects. Residue from the  
5 EMR agent can accumulate on the mold surface. The residue can obscure detailed features of the mold, thereby preventing proper imprinting of the features onto the surface of molded foam article. Also, the removal of the residue requires periodic cleaning of the mold. Periodic cleaning results in removing the mold from service, thereby affecting the service time of the mold.

10 Also, EMR agents can adhere to the surface of the molded foam article and is often removed from the mold surface with the molded foam article. The EMR agents must therefore be replaced to provide continued release of the molded foam articles. The necessity for repeated additions of EMR agent results in additional expense due to both time losses for applying additional quantities of EMR agent to the mold surfaces as well as the cost of the  
15 EMR agent itself.

The use of an internal mold release (IMR) agent can alleviate some of the problems associated with the use of an EMR agent alone. IMR agents can be included as integral components of a foamable reaction mixture. Examples of processes for preparing molded foams from a foamable reaction mixture, containing an IMR agent, are disclosed in U.S.  
20 Patent Nos. 4,201,847; 4,254,228; 4,868,224; 5,019,317; and 5,389,696. A foamable reaction mixture containing an organic polyisocyanate and an active hydrogen containing fatty acid ester is specifically disclosed in U.S. Patent Nos. 4,201,847 and 4,225,228. U.S. Patent No.

5,389,696 specifically discloses the use of an expensive mixture of fatty esters as an IMR agent.

Unfortunately, IMR agents do not generally function as well as EMR agents. Also, IMR agents can be expensive. Therefore, it would be desirable in the art of preparing molded foam articles to have an IMR composition that reduces the mold opening force required to free a molded foam article from a mold. It would also be desirable to have an IMR composition that reduces the amount of an EMR agent that needs to be applied to a mold's surface. Furthermore, it would be desirable to replace an IMR agent with a less expensive material without adversely affecting the opening force. Moreover, it would also be desirable to reduce the cycle time associated with the use of EMR agents.

#### SUMMARY OF THE INVENTION

According to the present invention, a method for preparing a molded polyurethane foam article using an internal mold release composition is provided. More specifically, a method using an IMR agent and an IMR-enhancer compound to prepare a molded polyurethane foam article is provided.

In its generic embodiment, the method comprises (a) forming a polyurethane-forming mixture by mixing (i) a fatty acid condensation product, (ii) an IMR-enhancer compound, (iii) an isocyanate, (iv) a polyol, (v) a catalyst, and (vi) a blowing agent; (b) filling a mold with the polyurethane-forming mixture; (c) forming a molded foam article; and (d) removing the molded foam article from the mold. The IMR-enhancer compound is any liquid petroleum product within the viscosity range of products called oils. A molded foam article prepared in accordance with the method is also provided.

In its preferred embodiment, the method comprises first mixing the fatty acid condensation product, the IMR-enhancer compound, and the polyol to yield an enhanced IMR "B" side composition, followed by admixing the other components and then performing steps b-d. In an alternate embodiment, the fatty acid condensation product and the IMR-enhancer compound can first be added to the isocyanate to yield an enhanced IMR "A" side composition.

In an alternate embodiment, a method for preparing an enhanced IMR composition comprising reacting a fatty acid condensation product with an isocyanate in the presence of an IMR-enhancer compound, wherein the fatty acid condensation product has at least one active hydrogen containing group, is provided. An enhanced IMR composition prepared in accordance with the method of the alternate embodiment is provided.

#### DESCRIPTION OF THE INVENTION

In the generic embodiment of the present invention, the method comprises (a) forming a polyurethane-forming mixture by mixing (i) a fatty acid condensation product, (ii) an IMR-enhancer compound, (iii) an isocyanate, (iv) a polyol, (v) a catalyst, and (vi) a blowing agent; (b) filling a mold with the polyurethane-forming mixture; (c) forming a molded foam article; and (d) removing the molded foam article from the mold.

In the present invention, polyurethane refers a polyurethane compound obtained by the reaction of a polyol with an isocyanate.

Fatty acid condensation products suitable for the practice of the present invention can be prepared from fatty acids and alcohols, amino alcohols, amines or mixtures thereof. Useful fatty acids include saturated or unsaturated fatty acids. Preferred are aliphatic acids.



200°C. The esterification reaction can optionally be conducted in a vacuum. Water can optionally be removed from the reaction mixture by azeotropic distillation.

A fatty acid condensation product is effective as IMR agent in amounts that allow removal of the molded foam article without destroying the article. An effective amount is in the range of from about 2 to about 15 wt %, based on the weight of the polyurethane-forming mixture. Preferably, the effective amount is within the range of from about 5 to about 10 wt%.

An IMR-enhancer compound of the present invention is any liquid product of petroleum within the viscosity range of products called oils. Examples of such oils are those commonly referred to as mineral oils. The preferred mineral oils are those known in the art as white mineral oils.

An IMR-enhancer compound is effective when it is added in amount sufficient to reduce the force to remove the molded foam article from the mold. The IMR-enhancer compound can be added in any amount within the range of from about 10 to about 100 wt % based on the weight of the fatty acid condensation product. Preferably, the IMR-enhancer compound is added in an amount within the range of from about 80 to about 90 wt%.

Any isocyanate or polyisocyanate known and used in the art of preparing molded foams can be suitable for the practice of the present invention, including aromatic and aliphatic isocyanates and polyisocyanates. The terms isocyanate and polyisocyanate are herein used interchangeably without limiting the scope of the individual terms. The isocyanate functionality can be present in an amount of from about 0.2 wt% to about 40 wt%.



U.S. Patent No. 4,785,027, incorporated herein by reference, discloses isocyanate compounds useful in practicing the present invention. Examples of useful isocyanates include the isomers of toluene diisocyanate (TDI) such as 2,4-toluene diisocyanate and 2,6-toluene diisocyanate, prepolymers of TDI, bis(4-isocyanatophenyl)methane (MDI),  
5 prepolymers of MDI, bis(isocyanatoethyl fumarate), dianisidine diisocyanate, toluidine diisocyanate, ethylene diisocyanate, 1,4-tetramethylene diisocyanate, 1,6-hexamethylene diisocyanate, 1,12-dodecane diisocyanate, cyclobutane-1,3-diisocyanate, 1,5-diisocyanato-3,3,5-trimethylcyclohexane, 2,4-hexahydrotoluene diisocyanate, 2,6-hexahydrotoluene diisocyanate, perhydro-2,4'-diphenylmethane diisocyanate, perhydro-2,6'-diphenylmethane  
10 diisocyanate, and mixtures thereof. Preferred isocyanate compounds include TDI, MDI, prepolymers of TDI, prepolymers of MDI, and mixtures thereof.

Any compound with multifunctional active hydrogen containing groups can be used in the practice of the present invention. The term "active hydrogen compound" refers to a compound that will react with an isocyanate to form an adduct between the active hydrogen  
15 containing compound and the isocyanate. Active hydrogen compounds can be described as compounds having functional groups that contain at least one hydrogen atom bonded directly to an electronegative atom such as nitrogen, oxygen or sulfur.

Polyols useful in practicing the present invention are generally known and are described in such publications as High Polymers, Vol. XVI, "Polyurethanes, Chemistry and  
20 Technology" by Saunders and Frisch, Interscience Publishers, New York, Vol. I, pp. 32-42, 44-54 (1962) and Vol. II, pp. 5-6, 198-199 (1964); Organic Polymer Chemistry by K. J. Saunders, Chapman and Hall, London, pp. 323-325 (1973); Developments in Polyurethanes,

Vol. I, J. M. Burst, ed., Applied Science Publishers, pp. 1-76 (1978); and Reaction Polymers, W. Gum, W. Riese, and H. Ulrich, Eds., Hanser Publishers (1992), incorporated herein by reference. Suitable polyols have molecular weights of less than about 6000.

Examples of such polyols include (a) alkylene oxide adducts of polyhydroxyalkanes;  
5 (b) alkylene oxide adducts of non-reducing sugars and sugar derivatives; (c) alkylene oxide adducts of polyphenols; and (d) alkylene oxide adducts of amines and amine polyols.

Alcohols and amines are examples of other active hydrogen compounds that are useful in practicing the present invention.

Examples of catalysts useful in practicing the present invention include tertiary amine  
10 catalysts such as triethylene diamine, N-methyl morpholine, N-ethyl morpholine, diethylethanolamine, N-coco morpholine, 1-methyl-4-dimethylaminoethyl piperazine, and bis(N,N-dimethylaminoethyl)ether. Amine catalysts are usually used in an amount of from about 0.1 to about 5, preferably from about 0.2 to about 2 parts per 100 parts of polyol formulation.

15 Organometallic catalysts are also suitable. Examples include organolead, organoiron, organomercury, organobismuth, and preferably organotin compounds. Most preferred are organotin compounds such as dibutyltin dilaurate, dimethyltin dilaurate, stannous octoate, stannous chloride and similar compounds. Organometallic compounds are usually used in an amount from about 0.05 to about 2.0 parts per 100 parts of active hydrogen containing  
20 compound formulation.

Blowing agents useful for practicing the present invention include any gas or any material that generates a gas under the conditions of the reaction of the active hydrogen



copolymers containing at least one polyoxyalkylene segment and one poly(dimethylsiloxane) segment.

U.S. Patent Nos. 4,868,224; 5,019,317; and 5,389,696, incorporated herein by reference, disclose examples of IMR agents which can be included in the "B" side of the present invention.

The present invention can also use crosslinkers. Examples of crosslinkers include diethanolamine and methylene bis(o-chloroaniline).

In its preferred embodiment, the method comprises first mixing the fatty acid condensation product, the IMR-enhancer compound, and the polyol to yield an enhanced IMR "B" side composition, followed by admixing the other components and then performing steps b-d of the generic embodiment. In an alternate embodiment, the fatty acid condensation product and the IMR-enhancer compound can first be added to the isocyanate to yield an enhanced IMR "A" side composition. A molded foam article is prepared in accordance with the methods of the present invention.

In another alternate embodiment, a portion of the fatty acid condensation product, a portion of the IMR-enhancer compound, and the isocyanate are first mixed to yield an enhanced IMR "A" side composition and the residual portion of the fatty acid condensation product, the residual portion of the IMR-enhancer compound, and the polyol are mixed to yield an enhanced IMR "B" side composition. Then, steps b-d are performed.

In an alternate embodiment, a method for preparing an enhanced IMR composition comprises reacting a fatty acid condensation product with an isocyanate in the presence of an IMR-enhancer compound, wherein the fatty acid condensation product has at least one active

hydrogen containing group. In this embodiment, an enhanced IMR composition is prepared in accordance with the method of the alternate embodiment. Moreover, the enhanced IMR composition is admixed during the "forming a polyurethane-forming mixture" of the generic embodiment of the present invention.

5 The embodiments described herein are given to illustrate the scope and spirit of the present invention. The embodiments herein will make apparent to those skilled in the art other embodiments that may also be used. These other embodiments are within the scope of the present invention. Thus, the scope of the invention should be determined by the appended claims and their legal equivalents, rather than by the embodiments given herein.

#### 10 EXAMPLES

The following examples merely exemplify various embodiments of the invention. It is understood that the following examples are provided to further illustrate the invention. They do not in anyway limit the scope of the present invention.

#### Table of Compounds

15 The following compounds were used in the preparation of the exemplified dispersions. The table provides the compound's generic name, its trade mark name, and its vendor.

Generic Name	Trademark	Vendor
propylene oxide polyol, 165 molecular weight	VORANOL™ 2025 Polyol	The Dow Chemical Company
propylene oxide polyol, 1000 molecular weight	VORANOL™ 230-112 Polyol	The Dow Chemical Company
propylene oxide polyol, 165 molecular weight	VORANOL™ 230-660 Polyol	The Dow Chemical Company

Generic Name	Trademark	Vendor
propylene oxide polyol initiated with ethylene diamine, 340 molecular weight	VORANOL™ RA640 Polyol	The Dow Chemical Company
polymethylene polyphenyl isocyanate containing 4,4'-methylene bisphenyl isocyanate	PAPI™ 27 MDI	The Dow Chemical Company
polymethylene polyphenyl isocyanate containing 4,4'-methylene bis-phenyl isocyanate	PAPI™ 94 MDI	The Dow Chemical Company
polymethylene polyphenyl isocyanate containing 4,4'-methylene bis-phenyl isocyanate	PAPI™ 95 MDI	The Dow Chemical Company
33% triethylene diamine in dipropylene glycol	Dabco 33LV	Air Products and Chemicals Inc.
methyl azanorbornane	Dabco AN10	Air Products and Chemicals Inc.
pentamethyl diethylene triamine	Polycat 5	Air Products and Chemicals Inc.
polyether-modified polysiloxane	Tagostab B8418	The Goldschmidt Company
polyether-modified polysiloxane	Tagostab 8427	The Goldschmidt Company
Complex ester of pentaerythritol, adipic acid, and oleic acid with low hydroxyl content	Loxiol G71S	The Henkel Corporation
polyethylene wax	Chem Tren 2004	Chem Tren
decaglycerol tertaoleate	Drewpol 10-4-O	Stepan
decaglycerol decaoleate	Polyaldol DGDO	Lonza Speciality Chemicals
tallamidopropyl dimethylamine	Shercodine T	Scher Chemicals
tallamidopropyl dimethylamine	Lexamine T-13	Inolex Chemical Company

Generic Name	Trademark	Vendor
tallaic salt of tallamidopropyl dimethylamine	Lexamine T-13T	Inolex Chemical Company
pentaerythritol oleate adipate	Inolex POA	Inolex Chemical Company

### **EXAMPLE NOS. 1-10**

For Example Nos. 1 - 10, molded foam articles were prepared. The molded foam articles were prepared with "A" and "B" side formulations. The IMR-enhancer compound was added to the "B" side formulations, in accordance with the preferred embodiment of the present invention.

A heated mold was used to form the molded form article. The mold cavity was made of aluminum and maintained at 120°F (49°C). The cavity was approximately 3" (7.62 cm) in diameter with a 1" (2.54cm) x 3° draw. The mold core was made of stainless steel and maintained at 150°F (66°C).

A single light coating of external mold release (Chem Tren 2004 polyethylene wax) was applied to the core. When closed, the mold had a gap of 0.17" ( cm). With the mold open, a 7"x7"x0.75" (17.8 cm x 17.8 cm x 1.9 cm) polyvinylchloride (PVC) coverstock sheet was placed over the cavity, and a one ounce (28.4 gm) glass mat was placed on top of the PVC.

The polyol formulation ("B" side) was mixed with the polymeric isocyanate ("A" side) in a desired ratio and poured onto the PVC sheet overlaid with glass mat. The mold was closed for 2 minutes, then opened and the molded foam article was removed by simply

pulling it off of the core.

The force required to remove the molded foam article from the mold was measured qualitatively. A value of 1 indicated that no force was required, 2 indicated that some force was required, 3 indicated that a lot of force required, 4 indicated that some type of tool  
5 required to loosen the molded foam article, and 5 indicated that the molded foam article strongly adhered to the mold and that the article could not be removed without damage to the molded foam article.

Successive quantities of the polyurethane-forming mixture were added to the mold until (1) a value of 4 was achieved or (2) at least twelve molded foam articles were prepared.  
10 Each set of quantities was formed into molded foam articles, which together make up an Example. The external mold release agent was not replenished between the successive additions. The release value was recorded through preparation of the twelfth article.

#### 1. Example Nos. 1 and 2

Example No. 1 was prepared using 45% PAPI™ 94 MDI, 45% PAPI™ 95 MDI, and  
15 10% Polyaldol DGDO on the "A" side. The "B" side formulation contained 60 parts VORANOL™ 2025 polyol, 30 parts VORANOL™ 230-112 polyol, 5 parts glycerine, 1.5 parts water, 2.0 parts Dabco 33LV, 0.5 parts Tagostab 8427, and 1.0 parts Polycat 5. The ratio of A:B was 1.8:1.

Example No. 2 was prepared using the same formulations as in Example No. 1,  
20 except (1) 2.0 parts Tagostab B8418 replaced the 0.5 parts Tagostab 8427 and (2) 14 parts mineral oil was added on the "B" side. The ratio of A:B was 1.5:1.



Articles for Example No. 1

Article No.	Release Value
1	1
2	1
3	1.5
4	2
5	3
6	4

Articles for Example No. 2

Article No.	Release Value
1	1
2	1
3	1
4	1
5	1.5
6	1.5
7	2
8	3
9	4

5 A comparison of Example Nos. 1 and 2 clearly shows the advantage of using mineral oil as an IMR-enhancer compound.

2. Example Nos. 3 and 4

Example No. 3 was prepared using 100% PAPI™ 27 MDI on the "A" side. The "B" side formulation contained 42 parts VORANOL™ 2025 polyol, 33.6 parts VORANOL™  
 10 230-112 polyol, 8.4 parts glycerine, 2.0 parts water, 1.5 parts Dabco 33LV, 2.82 parts oleic acid, 3.66 parts Lexamine T-13, 2.0 parts Tagostab B8418, 1.5 parts Polycat 5, 12.0 parts mineral oil, and 14.0 parts Loxiol G71S. The ratio of A:B was 1.2:1.

Example No. 4 was prepared using 100% PAPI™ 94 MDI on the "A" side. The "B" side formulation was the same as used in Example No. 3, except (1) tall oil acid replaced

WO 00/12607

PCT/US99/19458

oleic acid, (2) Shercodine T replaced Lexamine T-13, and (3) mineral oil was not added. The ratio of A:B was 1.35:1.

Articles for Example No. 3

Article No.	Release Value
1	1.5
2	1.5
3	1.5
4	1.5
5	1.5
6	1.5
7	1.5
8	1
9	1
10	1.5
11	1
12	1.5

Articles for Example No. 4

Article No.	Release Value
1	1
2	1
3	1.5
4	1.5
5	1.5
6	2
7	2
8	2
9	2
10	2
11	2
12	2

The data shows that the absence of mineral oil makes removal of the article from the mold more difficult.

3. Example No. 5

Example No. 5 was prepared using 100% PAPI™ 27 MDI on the "A" side. The "B" side formulation contained 42 parts VORANOL™ 2025 polyol, 33.6 parts VORANOL™

230-112 polyol, 8.4 parts glycerine, 2.0 parts water, 1.5 parts Dabco 33LV, 2.82 parts tall oil acid, 3.66 parts Shercodine T, 2.0 parts Tagostab B8418, 1.5 parts Polycat 5, and 12.0 parts mineral oil. The ratio of A:B was 1.35:1.

Articles for Example No. 5

Article No.	Release Value
1	1
2	1.5
3	2
4	4

5

The data indicates that mineral oil alone is not an effective internal mold release agent.

4. Example Nos. 6-8

Example No. 6 was prepared using 100% PAPI™ 94 MDI on the "A" side. The "B" side formulation contained 50 parts VORANOL™ 230-660 polyol, 30 parts VORANOL™ 230-112 polyol, 8.0 parts glycerine, 2.0 parts water, 1.5 parts Dabco 33LV, 2.0 parts Tagostab B8418, 1.5 parts Polycat 5, and 30 parts Loxiol G71S. The ratio of A:B was 1.3:1.

Example No. 7 was prepared using the formulations of Example No. 6, except the "B" side formulation also contained 14 parts mineral oil. The ratio of A:B was 1.15:1.

Example No. 8 was prepared using the formulations of Example No. 6, except (1) the Loxiol G71S was not added and (2) 14 parts mineral oil was added. The ratio of A:B was 1.45:1.

Articles for Example No. 6

Article No.	Release Value
1	1.5
2	1.5
3	1.5
4	1.5
5	1.5
6	1.5
7	1.5
8	1.5
9	1.5
10	1.5
11	1.5
12	1.5

Articles for Example No. 7

Article No.	Release Value
1	1
2	1
3	1
4	1
5	1
6	1
7	1
8	1
9	1
10	1
11	1
12	1

Articles for Example No. 8

Article No.	Release Value
1	1
2	1.5
3	2
4	4

5

This data shows the advantage of using a mineral oil as an IMR-enhancer compound.

### 5. Example Nos. 9 and 10

Example No. 9 was prepared using 100% PAPI™ 94 MDI on the “A” side. The “B” side formulation contained 50 parts VORANOL™ 2025 polyol, 40 parts VORANOL™ 230-112 polyol, 10 parts glycerine, 2.0 parts water, 8.34 parts Lexamine T-13T, 0.26 parts oleic acid, 2.0 parts Tagostab B8418, 1.5 parts Dabco AN10, and 30 parts Inolex POA. . The ratio of A:B was 1.2:1.

Example No. 10 was prepared using the formulations of Example No. 9, except (1) tall oil acid replaced oleic acid and (2) 14.0 parts mineral oil was added to the “B” side formulation. The ratio of A:B was 1.6:1.5.

10

#### Articles for Example No. 9

Article No.	Release Value
1	1
2	1
3	1
4	1
5	1.5
6	4
7	2
8	4
9	4

#### Articles for Example No. 10

Example No.	Release Value
1	1
2	1
3	1
4	1
5	1
6	1
7	1.5
8	2
9	2
10	3
11	4
12	4

This data shows the advantage of using an IMR-enhancer compound such as mineral

15 oil.

**What is claimed is:**

- 1 1. A method for preparing a molded foam article comprising:
  - 2 a. forming a polyurethane-forming mixture by mixing
    - 3 i. a fatty acid condensation product;
    - 4 ii. an IMR-enhancer compound;
    - 5 iii. an isocyanate;
    - 6 iv. a polyol;
    - 7 v. a catalyst; and
    - 8 vi. a blowing agent;
  - 9 b. filling a mold with the polyurethane-forming mixture;
  - 10 c. forming a molded foam article; and
  - 11 d. removing the molded foam article from the mold.
- 1 2. The method of Claim 1, wherein the fatty acid condensation product, the IMR-  
2 enhancer compound, and the isocyanate are first mixed to yield an enhanced IMR "A"  
3 side composition.
- 1 3. The method of Claim 1, wherein the fatty acid condensation product, the IMR-  
2 enhancer compound, and the polyol are first mixed to yield an enhanced IMR "B"  
3 side composition.
- 1 4. The method of Claim 1, wherein a portion of the fatty acid condensation product, a  
2 portion of the IMR-enhancer compound, and the isocyanate are first mixed to yield an  
3 enhanced IMR "A" side composition and wherein the residual portion of the fatty acid  
4 condensation product, the residual portion of the IMR-enhancer compound, and the

polyol are mixed to yield an enhanced IMR "B" side composition.

5. The method of Claim 1, wherein the fatty acid condensation product is a condensation product of a fatty acid selected from the group consisting of ricinoleic acid, oleic acid, alaidic acid, stearic acid, palmitic acid, linoleic acid, octanoic acid, coconut oil acids, tallow fatty acid, paraffin oxidation acids, and tall oil fatty acid and the IMR-enhancer compound is mineral oil.

6. A molded foam article prepared in accordance with the method of Claim 1.

7. A method for preparing an enhanced IMR composition comprising:

a. reacting a fatty acid condensation product with an isocyanate in the presence of an IMR-enhancer compound, wherein the fatty acid condensation product, having at least one active hydrogen containing group.

8. An enhanced IMR composition prepared in accordance with the method of Claim 6.

9. The method of Claim 1, wherein the components mixed in the "forming a polyurethane-forming mixture" step further comprise the enhanced IMR composition of Claim 7.

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION  
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification <sup>7</sup> :</b> <b>C08K 5/09</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 00/12607</b> <b>(43) International Publication Date:</b> 9 March 2000 (09.03.00)
<b>(21) International Application Number:</b> PCT/US99/19458 <b>(22) International Filing Date:</b> 26 August 1999 (26.08.99)  <b>(30) Priority Data:</b> 60/098,002      26 August 1998 (26.08.98)      US  <b>(71) Applicant (for all designated States except US):</b> THE DOW CHEMICAL COMPANY [US/US]; 2030 Dow Center, Mid- land, MI 48674 (US).  <b>(72) Inventor; and</b> <b>(75) Inventor/Applicant (for US only):</b> NISWANDER, Ron, H. [US/US]; 1902 Twin Tulip Court, Fresno, TX 77545 (US).  <b>(74) Agent:</b> TYLER, Gene, L.; Patent Department, B-1211, 2301 Brazosport Boulevard, Freeport, TX 77541 (US).		<b>(81) Designated States:</b> AU, BR, CA, CN, IN, JP, KR, MX, RU, TR, US, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> INTERNAL MOLD RELEASE FOR LOW DENSITY REACTION INJECTION MOLDED POLYURETHANE FOAM		
<b>(57) Abstract</b>  A method for preparing a molded polyurethane foam article using an internal mold release composition is provided. More specifically, a method using an IMR agent and an IMR-enhancer compound to prepare a molded polyurethane foam article is provided. The method comprises (a) forming a polyurethane-forming mixture by mixing (i) a fatty acid condensation product, (ii) an IMR-enhancer compound, (iii) an isocyanate, (iv) a polyol, (v) a catalyst, and (vi) a blowing agent; (b) filling a mold with the polyurethane-forming mixture; (c) forming a molded foam article; and (d) removing the molded foam article from the mold. The IMR-enhancer compound is any liquid petroleum product within the viscosity range of products called oils.		



USA/PCT

As a below named inventor, I hereby declare that

- (a) My residence and Citizenship are as stated below my name. My P O (mailing) address is the same as my residence unless otherwise stated.
- (b) I verily believe I am/we are the original, first and sole/joint inventor(s) of the subject matter that is embraced by and for which a patent is sought on the invention entitled: **INTERNAL MOLD RELEASE COMPOSITION FOR LOW DENSITY REACTION INJECTION MOLDED POLYURETHANE FOAM**

and the specification of which: ☐ is attached hereto (\_\_\_\_\_)   
 (check one) ☒ was filed on 26 August 1999 as (43050).   
 Application No PCT/US99/19458   
 and was amended on \_\_\_\_\_

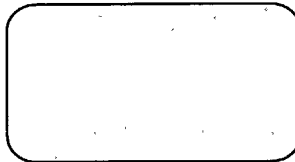
- (c) I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above
- (d) I acknowledge my duty under 37 CFR 1.56 to disclose to the U.S. Patent and Trademark Office all information known to me to be material to patentability as defined in 37 CFR 1.56(b). If this application is a continuation-in-part application, I acknowledge the duty to disclose all information known to me to be material to patentability as defined in 37 CFR 1.56(b) that became available between the filing date of the prior application from which priority is claimed in part (f) below, and the national or PCT international filing date of this application
- (e) I hereby claim foreign priority benefits under 35 U.S.C. § 119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate listed below or §365(a) of any PCT international application that designated at least one country other than the United States of America listed below, and also identify below any other foreign equivalent application for patent or inventor's certificate or any other equivalent PCT international application having a filing date before that of the application on which priority is claimed.

PRIOR FOREIGN APPLICATION(S)			PRIORITY CLAIMED	CERTIFIED COPIES INCL.
Number	Country or PCT	Day/Month/Year Filed		
<input type="checkbox"/>	Additional claims for benefit are attached.		<input type="checkbox"/>	<input type="checkbox"/>

- (f) I hereby Claim the benefit under 35 U.S.C. §119(e) of any United States provisional application(s) listed below, or under 35 U.S.C. §120 of any United States application(s), or under § 365(c) of any PCT international application designating the United States of America listed below:

<b>US or PCT Appl. Serial No.</b> <b>60/098,002 ✓</b>	<b>Filing Date</b> <b>26 August 1998 ✓</b>	<b>Status at Application Filing Date</b> <b>Abandoned</b>
--	---	--

I hereby appoint the attorney(s) and/or agent(s) at the following Customer No. to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith. Address all correspondence to The Dow Chemical Company's appointed counsel at



This appointment, including the right to delegate this appointment, shall also apply to the same extent it is applicable under the laws of the United States of America to any proceedings established by the Patent Cooperation Treaty.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under 18 U S C § 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Inventor(s)

At: Freeport, Texas 77451, USA  
this 28 day of March, 2001

Signature: Ron H. Niswander  
Full Name: Ron H. Niswander  
Residence: 1902 Twin Tulip Court  
Fresno, Texas 77545 TX.  
Country: United States of America  
Citizenship: United States of America ✓  
P. O. Address: Same as Residence

At: \_\_\_\_\_  
this \_\_\_\_\_ day of \_\_\_\_\_, 20\_\_\_\_

Signature: \_\_\_\_\_  
Full Name: \_\_\_\_\_  
Residence: \_\_\_\_\_

Country:  
Citizenship:  
P. O. Address:

At \_\_\_\_\_  
this \_\_\_\_\_ day of \_\_\_\_\_, 20\_\_\_\_

Signature: \_\_\_\_\_  
Full Name: \_\_\_\_\_  
Residence: \_\_\_\_\_

Country \_\_\_\_\_  
Citizenship \_\_\_\_\_  
P. O. Address \_\_\_\_\_

At. \_\_\_\_\_  
this      day of \_\_\_\_\_, 20\_\_\_\_

Signature: \_\_\_\_\_  
Full Name: \_\_\_\_\_  
Residence: \_\_\_\_\_

Country:  
Citizenship:  
P. O. Address:

☐ Additional names and signatures are attached.